

LED Based Soil Spectroscopy

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Abstract

Soil is a medium for plant roots to grow, absorb water and necessary solutes for growth. Soil macronutrient testing is helpful for determining the nutrient content in soil before applying fertilizer for quality and process controls of agricultural productivity and soil fertility. Spectroscopy is an emerging technology which is rapid and simple has been widely used in agricultural and food analysis processes. The capability of spectroscopy to characterize material from the transmission or absorbance has been used in this paper to measure nitrogen (*N*), phosphorus (*P*) and potassium (*K*) content in organic soil. The paper details preliminary characterization of soil spectroscopy with a Deuterium-Halogen lamp and spectrometer to measure the absorbance level of the macronutrients. The extracted nutrients were mixed with the colour reagent and specific coloured solution was developed. The result shows high absorbance level for *N* and *P* are at 970 nm in wavelength. In addition, *N* give absorbance at wavelength 450 nm and *P* yield absorbance at 800 nm wavelength. *K* was measured high at 620 nm. Further experiments were conducted to measure the absorbance characteristic of *N*, *P* and *K* for 20 minute period. The result shows that *P* and *K* has constant value of absorbance for 20 minutes duration while *N*, have stable absorbance value after 10 minutes being illuminated by 470 nm blue light-emitting diode (*LED*). For future works, the optical measurements will be implemented using visible and near infrared *LED* and the photodetector in order to replace the spectrometer usage for soil spectroscopy. This would lead to achieve the primary objective of this research in developing a simple and low cost spectroscopy uses *LED*.

Keywords: Spectroscopy; Agriculture soil; Absorbance level; Soil macronutrients; *LED*

1. Introduction

Soil macronutrients, nitrogen (*N*), phosphorus (*P*) and potassium (*K*) are essential for plant growth and needed in large quantity [1, 2, 3]. *N*, *P* and *K* are the most important nutrients in agriculture and *NPK* fertilizer is the most common fertilizers available in the market [3]. Excessive use of fertilizer can lead to surface and ground water pollution and also affect the quality of the products. The quantity of *N*, *P* and *K* varies depending on the type of crops and plant growth level. Therefore, the quantity of fertilizer has to be estimated based on the requirements for optimum production at each location in the field [1, 2].

Nowadays, soil nutrient testing or precision agriculture (*PA*) is required to determine the nutrients availability in soil before applying any fertilizer for quality and process control of agriculture produce and soil fertility [4]. One of *PA* is conventional soil testing which requires the

farmers to collect soil samples from their crop field and send to specialized soil analysis laboratory and wait for one to two weeks for the result. This method is time consuming, expensive, require highly skill operators and variables that affect the crop yield cannot be optimized at real-time [1, 4, 5]. Due to the cost, the number of samples analyse per field may be limited and cause the soil nutrient concentrations within the field cannot be characterized effectively [1].

There are four types of soil sensor have been used to measure various soil parameters which are mechanical sensor, optical sensor, electrochemical sensor and electrical and electromagnetic sensor. Mechanical sensor is capable to measure soil physical composition; optical sensor use visible and near-infrared wavelength to measure reflectance, absorbance and transmittance of soil [5, 6]; electrochemical sensor use ion-selective electrode or ion-selective field effect transistor to detect the nutrients by interact with nutrient ion in soil solution [1, 5, 6]; and electrical and electromagnetic sensor use electrical circuits to determine the soil texture, salinity, organic matter, moisture content and other parameters [5, 6]. According to Kulkarni *et. al* (2014), there are two suitable methods to measure the soil macronutrients which are optical method and electrochemical method.

Spectroscopy is a rapid, less expensive and non-destructive analytical technique [7]. It is widely use in optical method to detect the nutrients content in soil without the needs of chemicals such as laser-induced breakdown spectroscopy (*LIBS*) and near infrared (*NIR*) spectroscopy. According to the absorbance or reflectance of materials, spectroscopy technique can distinguish the elements corresponding its specific wavelength [7, 8]. One of the method which is *LIBS* technique give real-time analysis, simple sample preparation and small amount sample requirement but due to soil heterogeneity and matrix effect, this technique is difficult to analyze [4]. Further explanation is discussed in section 1.2.

To overcome *LIBS* technique limitation, *LED* and colour reagent has been implemented because this technique is less expensive and low power consumption [9]. This technique is adopted for this research not limited to its efficiency and low power consumption, but it does not require acids or other dangerous chemicals.

In this paper, the optical sensor method is chosen and an initial objective is to study the absorption of soil sample using visible light and obtain the optimum wavelength that shows the maximum absorbance for soil sample. *LED* light sources in visible range (470 nm to 700 nm) and near infrared wavelength range (735 nm to 1500 nm) will be used in future works.

1.1. Transmittance and Reflectance of Light

Each molecule has its own unique absorption and reflection spectrum. For soil nutrients detection, Beer-Lambert Law is utilized. Beer-Lambert law stated that there is a relationship between absorbance and concentration of an absorbing species in a solution and path length [10, 11].

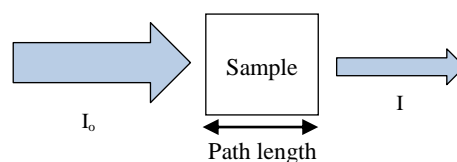


Figure 1 Absorption of light by a sample

Figure 1 shows the light absorption by a sample when light passes through it. The measurements are usually made in terms of transmittance (*T*) and absorbance (*A*) which can define as

$$T = \frac{I}{I_0} \quad (1)$$

$$A = -\log T = -\log \frac{I}{I_0} = \epsilon LC \quad (2)$$

Where I_0 = initial light intensity

I = light intensity after passing through the sample.

ε = molar absorptivity ($\text{L mol}^{-1} \text{cm}^{-1}$),

L = path length (cm) and

C = concentration of the absorbing chemical species (mol L^{-1}) [10, 12].

The significance of Beer-Lambert law is to measure the absorbance of a particular sample and to infer the concentration of the solution. There are two situations need to be considered. First, if a light beam of appropriate wavelength passes through a diluted solution, the photons will encounter a small number of absorbing chemical species (chemical species will absorb light at a particular wavelength) and the result will be obtained high T and low A [12]. Second, if the beam of light encounter diluted solution for a long period of time, the result will be encountered low T and high A . Thus, Beer-Lambert law stated that absorbance is proportional to the concentration of the sample and proportional to the path light of a beam through the sample [12, 13].

1.2 Laser Based Spectroscopy

Laser-induced breakdown spectroscopy (*LIBS*) is based on atomic emission spectroscopy that has been used widely for soil analysis. This method uses laser as the light source. The high power pulsed laser beam is focused on the surface of a solid sample to form microplasma. The microplasma will emit light characteristics of the elemental composition of the sample. The emitted light is collected by fiber and detected to monitor the concentrations of nutrients via their unique spectral signature [4, 14, 15]. During calibration phase, *LIBS* provides quantitative measurements and suitable for low cost field portable instrumentation [14]. This method is widely used to detect the total carbon content and contaminations in polluted soil. The advantages of this method are real-time analysis, high sensitivity, multi-elemental detection of major and trace elements require minor sample preparation and small requirement but the drawback is due to soil heterogeneity and matrix effect [4, 15].

1.3 LED Based Spectroscopy

LED based spectroscopy uses *LED* as light source and spectrometer to measure the light spectrum after passing through a sample. The soil nutrients N , P and K are verified using standard soil test kit which available in the market along with a standard colour chart [2]. The reagent in test kit is mixed with the nutrients in soil and develops coloured solution that gives different colour intensity depending on the amounts of available nutrients [16]. Using standard colour chart, the result is not reliable because the result always fluctuating due to tester's judgments. Therefore, spectrometer is used to investigate the colour-develop solution. Solution of tested soil is illuminate by visible and near-infrared wavelength range to measure the absorption peak to choose the suitable *LED* [9]. The selected *LED* is then illuminating the solution and lights will be reflected depending on the absorbance coefficient of the soil. The reflected light is received by paired optical fiber cable and send to the spectrometer to determine the intensity of transmitted light [2, 9]. When light travels through the solution, intensity of light decreased with the increasing number of absorbing species in the solution [9].

LEDs are highly suitable as a light source due to wide wavelength range from ultraviolet to infrared, less expensive and have long durability. Therefore, it is possible to find matching *LED* wavelength of the absorption band of sample nutrients. The emission spectra of the *LEDs* are measured with an optical spectrum analyzer. The quantitative analysis of three soil nutrients is performed using colour reagent solution [9].

In this paper, the characterization of macronutrient content of organic soil sample is reported using visible light wavelength range using light source Deuterium-Halogen lamp (*DH*) and blue *LED*. The result from this paper would help to develop a simple and low cost spectroscopy using *LED* in determining the macronutrients content in various types of soil samples.

2. Methodology

Figure 2 shows the experimental setup to measure the absorbance level of organic soil. The experimental setup consists of three parts, light source, sample and spectrometer. For the initial experiment, light source used was Deuterium-Halogen lamp (*DH*) with a wavelength range from 210 nm to 1200 nm and couple to the samples in a cuvette. The data was send to Ocean Optic spectrometer which has a range from 200 nm to 1100 nm and a spectral resolution to 0.65 nm. The spectrometer was interfaced to the computer using Ocean View software. Ocean View is a specifically designed program provided by Ocean Optics in order to acquire the data from the spectrometer in real time. In this experiment, optical attenuator was used to reduce the light source intensity. Thus, by having the optical attenuator, the power dissipation can be controlled. Further experiment was conducted using 470 nm blue *LED* by replacing *DH* lamp to measure the absorbance level of soil macronutrients.

The organic soil sample has been oven dry at temperature of 50°C for 12 hours. The soil nutrient extraction is using NPK soil test kit which is available in the market. NPK soil test kit senses the amount of *N*, *P* or *K* in soil samples and depending on the amount of the elements in the soil, the colour of solution changes [16]. Experimentally, the extraction of the nutrients on the tested soil was done. Then, the extracted solution was divided into three test tubes and mix with provided extractants for *N*, *P* and *K*. The developed colour solution of the mixture was compared with colour chart provided. The colour-develop solution was dripped into quartz cuvette to measure the absorbance level of the nutrients.

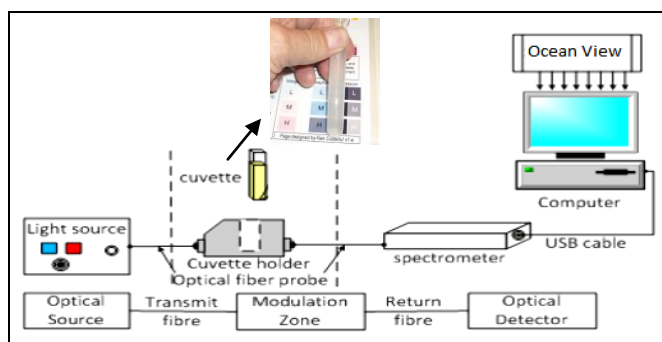


Figure 2 *DH* experimental setup

3. Result and Discussion

Using extractants, the colour-develop solution produce low red colour for *N*, medium blue colour for *P* and white colour for *K*. From the colour chart provided, *N* give low concentration value, medium concentration for *P* and high concentration for *K*. Further analysis using spectrometer was conducted.

Table 1 shows the tabulated result for concentration of *N*, *P* and *K* for experimentation and as reported by Ramane *et. al* (2015), Singh *et. al* (2014) and Benton (2012). These results are based on extracted nutrients using colour reagent and a colour chart to measure the concentration of nutrient ions in the colour-developed solution. From the conducted experiment, the concentration of *N*, *P* and *K* are in the same agreement with Ramane *et. al* and only *K* give same agreement with Singh *et. al*. According to Benton (2012), the approximate concentration of plant elements for healthy plant growth has obtained high *N* concentration, low *P* concentration and medium *K* concentration. However, the result of the experiment gives different concentration value. There are many possible reasons why the results differ from another researcher such as due to the types of soil, sample preparation procedure, particle size of the sample and the colour of the soil.

Figure 3 indicates the plotted graph of absorbance level for organic soil. The wavelength range selection will be from 400 nm to 1200 nm. Wavelength below 400 nm will not be considered. From the plotted graph, *N* and *P* give absorption peak at same wavelength of 970 nm. Moreover, *N* give high absorbance around 450 nm, *P* also gives absorption peak at 800 nm. In addition, *K* yield highest absorption peak at wavelength 620 nm. Table 2 shows the wavelength of *N*, *P* and *K* for visible light wavelength range for experimentation and as reported by another researcher.

Table 1 Comparison of concentration level with other researchers (L: Low, M: Medium, H: High) [2, 16, 17]

Nutrient	Concentration			
	Experimental	Ramane <i>et. al</i>	Singh <i>et. al</i>	Benton
Nitrogen	L	L	M	H
Phosphorus	M	M	L	L
Potassium	H	H	H	M

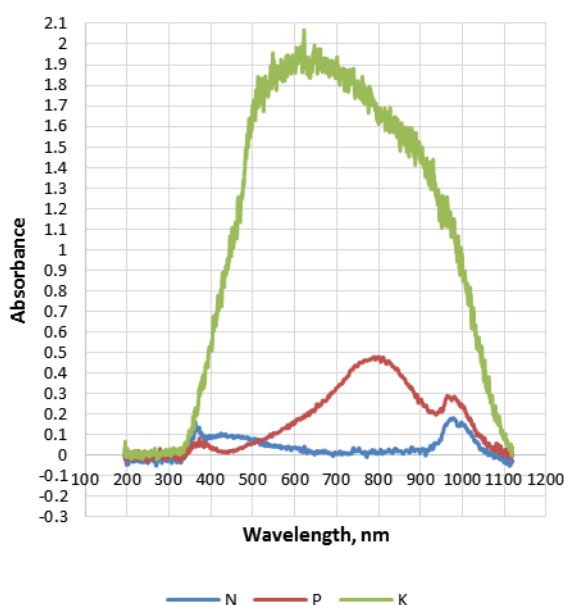


Figure 3 Absorbance level of organic soil

Table 2 Comparison of peak wavelength value with other researchers [9, 14]

Nutrients	Wavelength, nm		
	Experimental	Yokota <i>et. al</i>	Hussain <i>et. al</i>
Nitrogen	450	540	
	970	650	
Phosphorus	800	700	255
	970	900	
Potassium	620		404

From the result obtained, the wavelength of absorbance gives same agreement with Yokota *et. al* (2007) and Hussain *et. al* (2007) for *P* but not for *N* and *K*. This is may be due to calibration setup error, different composition of chemical reagent and the type of soil.

Further experiment was conducted using 470 nm blue LED. Figure 4 shows the plotted graph of the absorbance level of organic soil for duration of 20 minutes at wavelength of 470 nm. The solution is placed in the cuvette after the extracted solution develop specific colour and the absorbance of the solution is recorded every two minutes for 20 minutes. Absorbance level of *P* and *K* give a constant value throughout the duration meanwhile, *N* has fluctuated in absorbance for the first 8 minutes. In the last 10 minutes, the value of absorbance for *N* almost stable.

From Table 1, the experimental data for concentration of *N*, *P* and *K* are low, medium and high respectively. According to equation 2 and Beer-Lambert Law, when a beam passing through a diluted solution, the absorbance will be low and when passing through highly concentrated solution, the photon will encounter a large number of chemical absorbing species and causing absorbance to be high [12]. From Figure 4, *N* has absorbance of 0.1 and *P* has absorbance around 0.15. Meanwhile, *K* has absorbance between 1.25 and 1.4. This result follows the Beer-Lambert Law which is higher concentration has higher absorbance value.

Theoretically, faster reaction between sample and reagent will produce less time in the absorbance versus time curve. The time is being affected by the particle concentration. The higher the concentration, the faster the absorbance of photon occur inside the sample solution because the presence of abundance of chemical absorbing species [19]. Reference [18] and [19] reported that lower concentration level will have slower time to reach its maximum absorbance compared to higher concentration. Since the concentration of *N* is low, it supposed to have longer time to reach maximum absorbance while *K* will have a shorter time to achieve maximum absorbance.

In future work, the absorbance and concentrations measurement data collection will be taken in shorter (around one minute) and longer duration (around one hour) to ensure the macronutrient achieve its maximum absorbance.

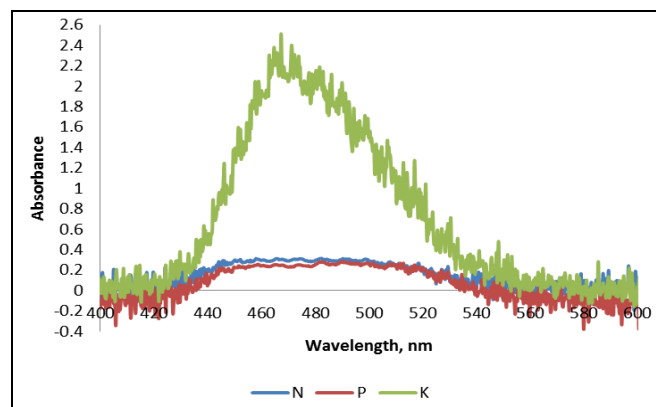


Figure 4 Absorbance level of sample for 20 minutes duration

4. Conclusion

This paper highlights the preliminary experimental analysis using a simple spectrometer for measuring absorbance of soil nutrients level. The extracted solution develops specific colour solution for different nutrients and colour intensity of the solution varies according to its nutrient concentration. From colour chart, soil sample has low concentration of *N*, medium for *P* and *K* has high concentration. Using the spectrometer, the wavelength of absorbance for *N* are 450 nm and 970 nm, *P* at 800 nm and 970 nm while *K* only at wavelength of 620 nm. These wavelengths give same agreement with Yokota *et. al* and Hussain *et. al* for *P* but not for *N* and *K* due to type of soil or different chemical reagent. Further experiment was conducted using blue LED. After 10 minutes being illuminated by LED, the absorbance

level for N , P and K are stable. In future, the visible and near infrared *LED* and photodetector will be implemented to replace the spectrometer for soil spectroscopy. This would help in developing a simple and low cost using *LED* based soil spectroscopy to achieve the primary objective of this research. In future, the sensing system with suitable *LED* for each nutrient will be conducted.

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